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Influences of Glass-to-Metal Sealing on the Structure and Magnetic Properties of an Fe/Co/V Alloy

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Apparent changes in the coercivity and remanence of the magnetic reed material used in the remreed sealed contact were encountered during the glass-to-metal sealing operation. By using primarily metallographic observations correlated with magnetic data, it was determined that these changes were due to a modification of the 600°C-aged microstructure caused by the time/temperature cycle experienced in sealing. The room-temperature stable precipitates that are developed by the 600°C anneal to produce magnetic hardening are dissolved, and the alloy converts to a two-phase duplex body-centered cubic (BCC) microstructure along one-half to two-thirds of the reed shank. This produces a substantial increase in coercive force and some decrease in remanence. The structure and properties of the alloy before sealing have little influence on the reaction.

I. INTRODUCTION

A recent effort in the telecommunications industry has been the development of a remanent reed, sealed contact (remreed).[1] One problem encountered in this development was an apparent change in the magnetic properties of the Remendur (Fe, Co, V alloy) reed during the glass-to-metal sealing operation. During sealing, the reed can reach peak temperatures in excess of 1050°C and may be at temperatures in excess of 900°C for as long as 8 to 10 seconds. Preliminary data supplied to the authors indicated an increase in the coercivity of reeds from the sealing operation,[2] whereas the results of Kitazawa, Oguma, and Hara showed a 22-percent decrease in coercivity at switch sealing.[3] Therefore, a description of the exact nature of this change and an explanation of the mechanism by which it occurs were sought. Also, information on the possible influence of variations in the time/temperature sealing cycle on the magnitude of this change was considered relevant to determine if improvements could be achieved by such modifications.

This paper summarizes the results of both a laboratory study on the Remendur alloy and evaluation of manufactured contacts to ascertain the mechanism for the reported property changes. The normal sealing operation was also modified by increasing the sealing speed and reducing the sealing-lamp temperature to obtain data on possible modifications to minimize the changes. Also presented are data on the influence of microstructure from the prior strand-anneal and 600°C-aging anneal-heat treatments on the stability of the aged magnetic properties.

II. EXPERIMENTAL PROCEDURES

The evaluation on actual sealed reeds was carried out on Western Electric Company assembled contacts. A group of reeds stamped from a single coil of commercially melted and processed 0.535-mm Remendur wire was aged at 610°C in hydrogen for two hours. Some reeds of this group were assembled into contacts at two separate manufacturing facilities. Samples of the aged reeds and reeds that had been removed from these assembled contacts were supplied to the authors for evaluation (Experiment A). Additionally, contacts that had been sealed at an accelerated speed were provided (Experiment B). Finally, a two-group sample was supplied consisting of six typical contacts and six contacts sealed with a 100-W reduction in power on the sealing lamps (Experiment C).

The laboratory study was carried out on a selection of coils of wire from another commercial melt of Remendur (48.7-percent Fe, 48.2-percent Co, 2.90-percent V by weight with balance impurities Mn, Ni, C, S). The wire was supplied in a 900 to 950°C strand-annealed and quenched condition. Samples from the front and back ends of every coil from this melt were examined metallographically. A variety of microstructural variations were detected in this annealed wire. These ranged from a nearly-all-α_1 phase structure produced by an anneal at the lower extreme in the temperature range to an all-α_2 phase structure produced by an anneal at the upper temperature extreme.[4,5] A sample of 15 front and back coil ends covering this range in structures of the 0.535-mm strand-annealed wire was selected. The wires were aged at 620°C for three hours in an argon/10-percent hydrogen-gas mixture. Each of these coil ends was then divided into three separate lengths of wire. Each wire length was subjected to one of three different high-temperature exposures. These exposures were carried out by inserting the wires for a fixed length of time into an open-air muffle furnace set for a fixed elevated temperature. They were gripped in asbestos-covered tongs for the insertion to prevent the tongs from acting as a heat sink. The three time-temperature cycles used on the wires are shown in Figs. 1, 2, and 3. These profiles were produced by insert-

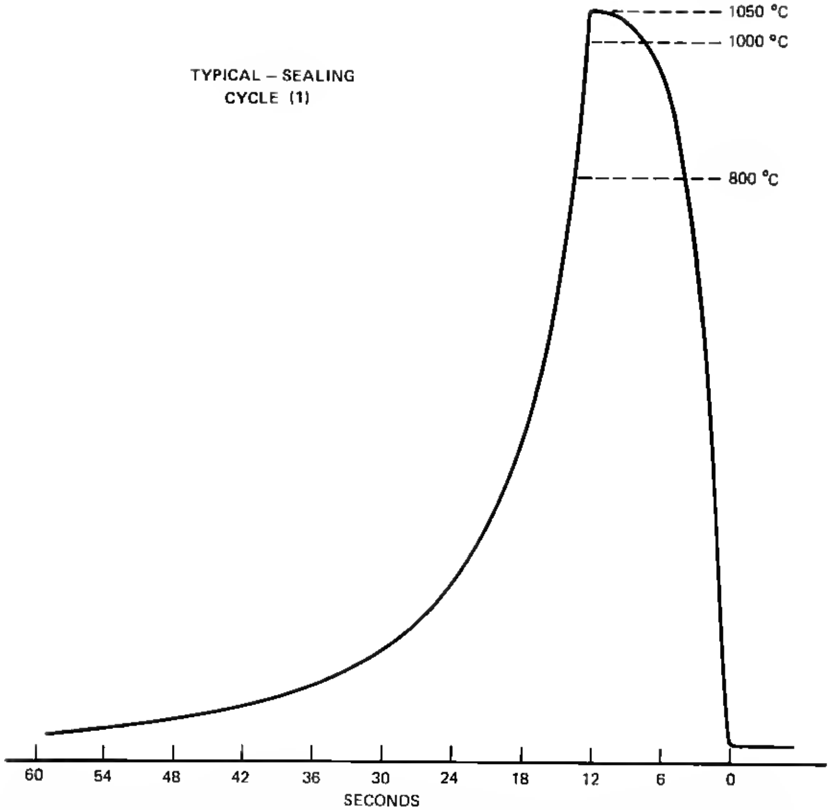

Fig. 1—Time/temperature thermocouple response for a 12-s insertion into a 1200°C furnace.

ing a chromel-alumel thermocouple made from 0.535-mm wire into the furnace in the same manner as the Remendur wire samples and monitoring the response on a strip-chart recorder. These cycles were selected to nearly duplicate typical sealing, reduced-temperature, and extended-time cycles, respectively. The typical cycle was determined from a previous study on the sealing of 237-type ferreed contacts.[6]

The production reeds and the laboratory samples in each heat-treated condition were evaluated metallographically and the magnetic parameters were determined. Metallographic preparation was routine, using a 5-percent Nital etch for 15 to 30 seconds. Structures were observed by optical light microscopy at 1500 magnification using Nomarski Differential Interference Contrast (DIC). All magnetic data were provided to the authors by E. C. Hellstrom. Coercivity and remanence were measured from full-loop traces on samples magnetized

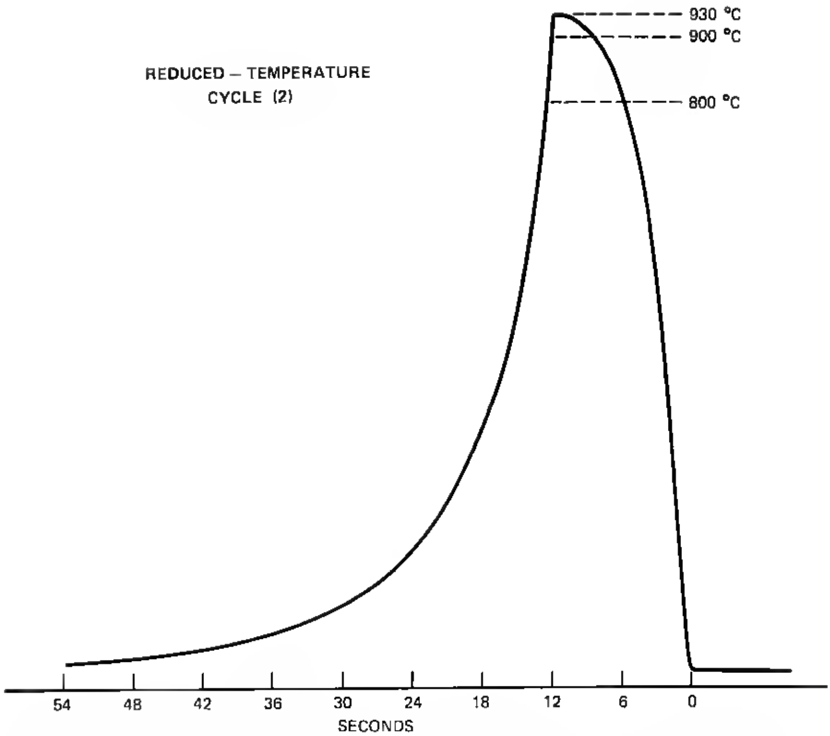

Fig. 2—Time/temperature thermocouple response for a 12-s insertion into a 1050°C furnace.

by an applied saturation field of 300 oersteds. All magnetic values presented are the average of multiple samples. The laboratory samples were 2.5 cm in length, and the production samples were actual reeds possessing their characteristic geometry (Fig. 4). The reeds were measured both as complete reeds with the search coil centered over the paddle portion and also as dissociated paddles and shanks.

III. RESULTS AND DISCUSSION

Microstructural comparisons and correlations were obtained on longitudinal sections of the shanks. Figure 5 shows the microstructures present in a typical reed shank after sealing, at approximately 1-mm increments along its length. It is apparent that, in this case, beginning at the paddle end, approximately the first 5.5 mm have undergone a structural change. This represents approximately two-thirds of the shank length. This heat-affected region is substantially larger than that actually encompassed by the glass in the seal.

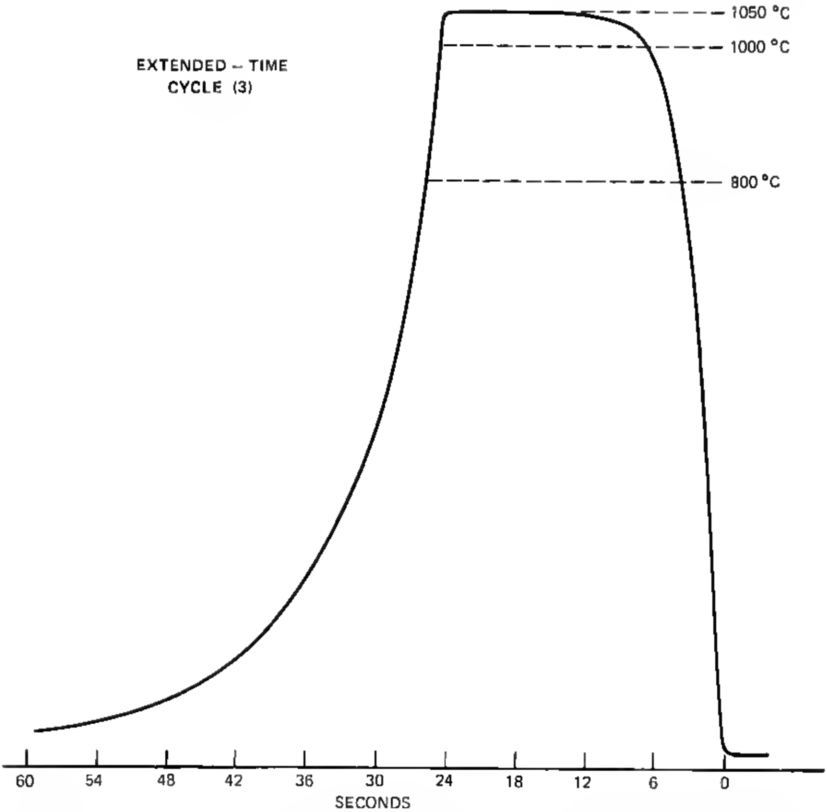

Fig. 3—Time/temperature thermocouple response for a 25-s insertion into a 1200°C furnace.

3.1 Laboratory-simulated sealing-cycle exposures

To determine the significance of this structural change, the laboratory experiment utilizing various elevated-temperature exposures was carried out. The magnetic-properties data for the 15 selected coils are summarized in Table I. Of the 15 coils selected, 11 are considered to possess a microstructure at least approximating a nearly equal distribution of the two phases, $\alpha_1 + \alpha_2$, in this duplex BCC structure.[7] This distribution is expected from an optimized-temperature strand anneal and quench.[4] In this strand-annealed state, these typical wires indicate a coercive force of 41 to 47 oersteds and a remanence of 21 to 23 maxwells for the specified measuring conditions. Three samples designated 208B, 240B, and 268B were strand-annealed at the low temperature extreme, i.e., less than about 900°C, and have a nearly single-phase structure of α_1 (BCC). They have a significantly lower co-

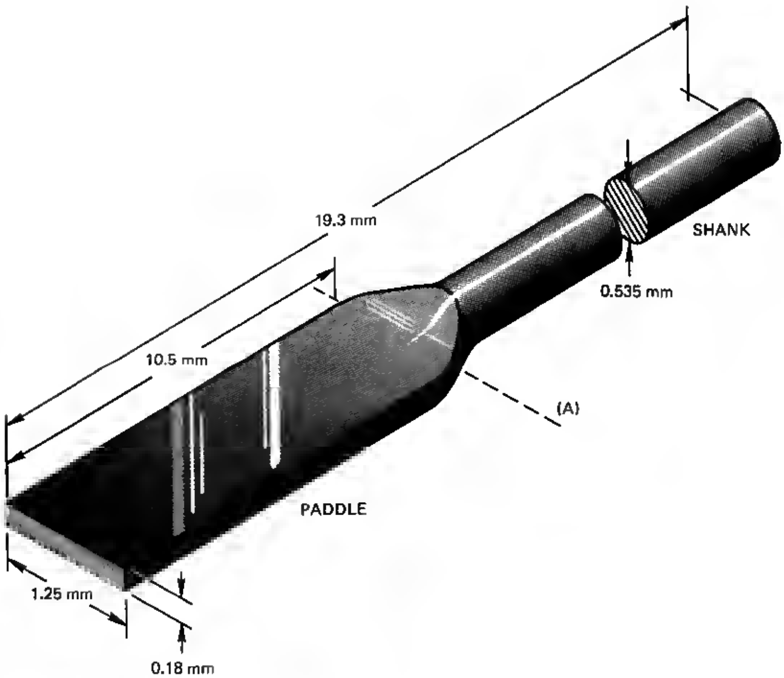

Fig. 4—Reed geometry used for magnetic evaluation.

ercive force with little difference in remanence when compared to the two-phase strand-annealed structure before aging. Finally, one sample designated 223B was strand-annealed at the upper temperature extreme, i.e., greater than about 950°C, and has a nearly single-phase structure of α_2 [the vanadium-supersaturated, FCC (face-centered cubic) to BCC transformation phase].[7] It shows not only a modestly reduced coercive force but a significantly lower remanence when compared to the two-phase strand-annealed structure. Representative microstructures for these three conditions are shown in Fig. 6, column 1.

Again referring to Table I, it appears that the 620°C-aging anneal has a tendency to normalize the magnetic properties. This is primarily a consequence of the precipitation of stable γ (FCC) phase,[6] relief of the $\gamma \rightarrow \alpha_2$ transformation strains,[7,8] and ordering of the α_1 matrix.[9] The four high- and low-temperature strand-annealed coils are still below the mean for the two-phased strand-annealed coils of 24 to 25 oersteds in coercive force and of 26 to 28 maxwells in remanence after aging. Representative microstructures in the 620°C-aged condition are also given in Fig. 6, column 2.

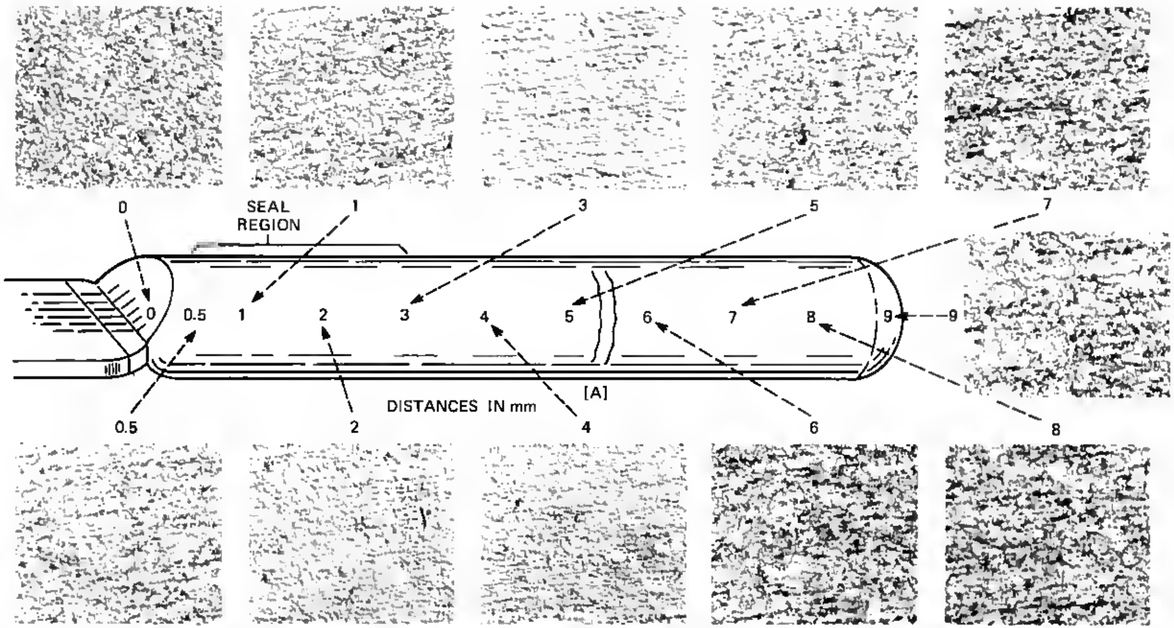

Fig. 5—Microstructures along the length of a typical sealed reed shank—etched 5-percent nital, DIC, 1500X. [A] represents the approximate end of the heat-affected zone.

Table I — Magnetic properties of Remendur wire before and after simulated sealing cycles 1, 2, and 3

Hc (oersteds)

Sample	Strand-Anneal Structure	Strand-Annealed	Aged 620°C—3 hr	Typical Cycle (1)	Low T Cycle (2)	Long t Cycle (3)
205 T	2 phase	41.4	24.3	32.8	16.6	28.6
205 B	2 phase	40.9	22.2	35.6	22.7	27.5
208 B	Primarily α_1	29.9	17.3	34.3	13.9	27.8
218 T	2 phase	46.6	25.5	35.4	—	29.1
218 B	2 phase	44.7	25.0	36.1	16.0	26.5
223 T	2 phase	42.6	23.8	37.4	19.7	28.8
223 B	Primarily α_2	35.5	22.7	33.0	14.2*	27.7*
231 T	2 phase	45.4	25.6	38.5	16.9	25.9
231 B	2 phase	47.3	27.3	38.5	18.3	28.9
236 T	2 phase	38.5	24.3	37.1	—	—
236 B	2 phase	43.5	26.4	36.3	14.5	26.3
240 T	2 phase	44.6	25.8	37.3	17.1	28.3
240 B	Primarily α_1	23.0	19.0	37.3	13.9	29.2
268 T	2 phase	46.7	27.0	38.5	17.8	27.4
268 B	Primarily α_1	21.0	19.0	37.0	—	29.5

$\emptyset_R$ (maxwells)

Sample	Strand-Anneal Structure	Strand-Annealed	Aged 620°C—3 hr	Typical Cycle (1)	Low T Cycle (2)	Long t Cycle (3)
205 T	2 phase	22.2	27.2	16.3	21.9	11.1
205 B	2 phase	22.3	23.2	18.1	24.2	11.3
208 B	Primarily α_1	22.1	21.4	16.5	20.6	11.0
218 T	2 phase	22.9	27.9	17.3	—	12.9
218 B	2 phase	22.6	27.5	18.2	22.6	11.8
223 T	2 phase	22.3	25.2	19.1	21.8	12.2
223 B	Primarily α_2	17.3	25.2	15.1	9.3*	7.6*
231 T	2 phase	22.8	28.3	20.2	22.9	11.3
231 B	2 phase	22.9	28.4	20.2	24.6	13.4
236 T	2 phase	21.9	27.7	19.6	—	—
236 B	2 phase	22.7	26.6	19.1	19.5	12.0
240 T	2 phase	22.7	25.7	19.0	23.0	11.4
240 B	Primarily α_1	20.6	20.8	19.5	19.3	12.2
268 T	2 phase	23.1	27.4	20.6	22.9	11.2
268 B	Primarily α_1	20.3	21.2	19.6	—	11.9

Material: 2.90 weight percent V (*i*) T = front of coil, (*ii*) B = back of coil, (*iii*) H_{app} = 300 oersteds, (*iv*) Values average of two or three samples, (*v*) Sample length = 2.5 cm.

* Aged at 515°C prior to elevated temperature exposure; 1.6-cm sample length.

Samples were exposed to the typical-sealing cycle given in Fig. 1, which was based on a previous experimental evaluation of the time-temperature cycle experienced by a 52-alloy reed in the sealing of a type 237 (nonlatching, Ni-Fe) reed contact.[6] This cycle creates an exposure to a peak temperature of 1050°C and temperatures in excess

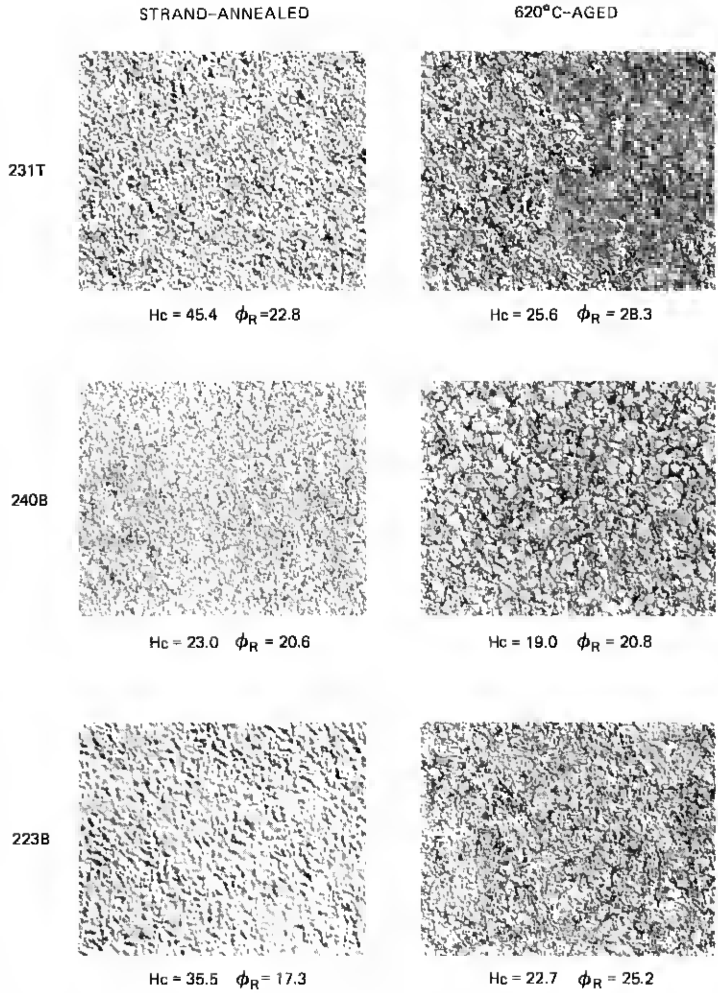

Fig. 6—Microstructures in strand-annealed and 620°C-aged conditions for two-phase (231T), primarily α_1-phase (240B), and primarily α_2-phase (223B) strand-annealed structures—etched 5-percent nital, DIC, 1500X.

of 900°C for approximately 7 seconds. This exposure produces a marked increase in coercive force into the range of 33 to 38 oersteds and a drop in remanence to 15 to 20 maxwells (Table I) for all samples. Representative microstructures given in Fig. 7, column 1, indicate that this change is a result of the material altering to the structure typically produced by the 900 to 950°C strand-anneal.[4] That is, the γ (FCC) precipitates are dissolved and portions of the matrix return to the *high-temperature* FCC phase and again undergo the FCC $\rightarrow$ BCC

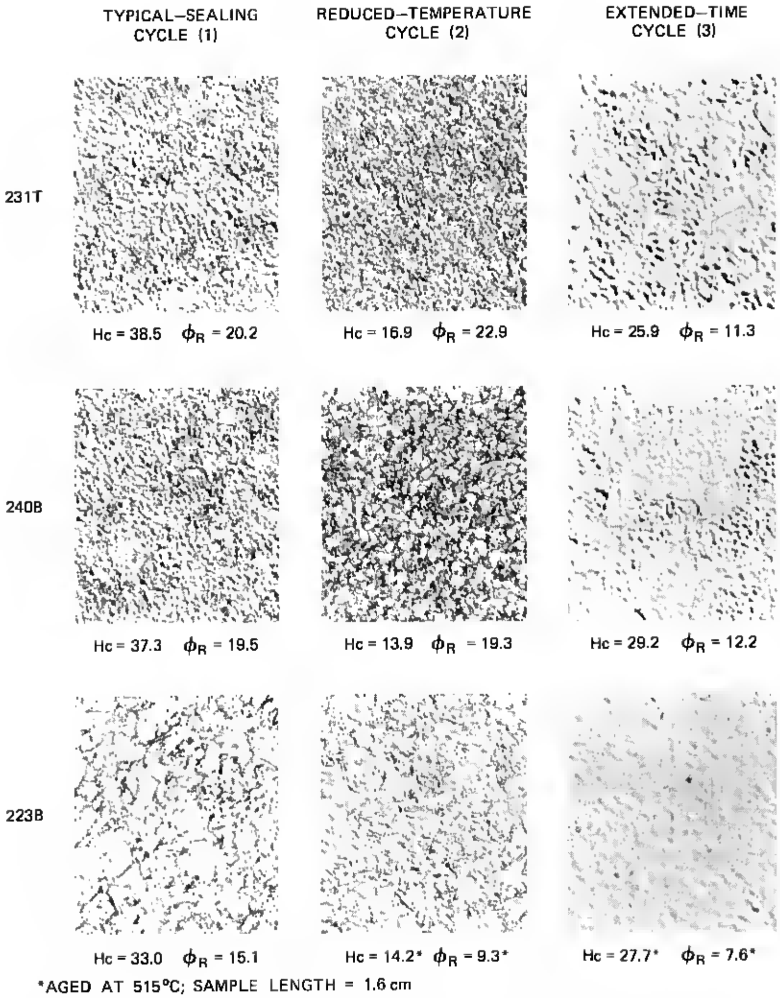

Fig. 7—Microstructures following various elevated-temperature exposures on samples shown in Fig. 6—etched 5-percent nital, DIC, 1500X.

transformation on cooling. This produces a highly strained lattice and, hence, the increased coercive force values. Similar results were obtained by Mahajan and Olsen[10] by exposing 600°C-aged Remendur to temperatures of 850, 950, and 1050°C for 5 seconds and measuring the magnetic changes as well as observing the structural variations utilizing transmission electron microscopy.

A slightly reduced temperature cycle was attempted to ascertain if a modest reduction in sealing temperatures could alleviate this change. This cycle, given in Fig. 2, had a peak temperature of 930°C and a temperature in excess of 900°C for less than 4 seconds. Magnetic-

properties results (Table I) show that the coercive force and remanence are both *reduced* by this cycle. However, the remanence decrease is small in all cases and, in general, those samples that had a two-phase strand-annealed microstructure before aging and high temperature exposure show a lesser decline in coercivity than the single-phase strand-annealed structures. Microstructural observations (Fig. 7, column 2) indicate that these changes are a result of the beginning of the dissolution of the γ (FCC) precipitate. This cycle is sufficiently low in temperature and time such that none of the matrix is returned to FCC structure at temperature and, hence, does not undergo the FCC $\rightarrow$ BCC transformation on cooling to markedly increase the magnetic hardness as observed in cycle 1.

To clearly prove that a reversion to the strand-annealed structure during sealing is the cause for the magnetic property changes observed in reeds,[2] an extended-time-at-temperature cycle was produced (Fig. 3). The peak temperature was maintained at 1050°C, but the time in excess of 900°C was increased to 20 seconds. In all cases, the structure totally converted to a single-phase α_2 matrix (Fig. 7, column 3), which is typical of a strand anneal at too high a temperature ($>$950 to 975°C).[4] These structures may be compared to the initial structure of coil 223B in this experiment (Fig. 6, column 1), which was stated to be a nearly-all-α_2 matrix. This heat treatment produces an extremely skewed hysteresis loop with a coercive force of 26 to 29 oersteds and a greatly reduced remanence to 11 to 13 maxwells (Table I) as measured on 1-inch samples. It is thus clear that very brief exposure to temperatures in excess of 900 to 1000°C can markedly alter the magnetic properties previously developed by the 600°C-aging heat treatment in Remendur.

3.2 Analysis of production reeds

The results of the before-and-after glass-to-metal sealing analysis on actual production reeds (Table II) correlate well with the above observations on the simulated-sealing temperature exposures. Listed are the coercive force and remanence as measured on the complete reed, with search coil on the paddle, and also as measured on the dissociated paddles and shanks both before and after exposure to sealing (Experiment A). As was previously observed by others,[2] the major effect of sealing is a marked increase in coercive force on the shank. In this instance, an average increase in the shank of about 6 oersteds from 23.5 to 29.5 occurred. As expected, no change in the paddle portion was observed, since it does not experience the peak temperature range.

The structural changes observed in the shanks of actual sealed reeds are very similar to those produced by the typical sealing cycle in the elevated-temperature-exposure experiments. The disappearance of the

Table II — Magnetic properties of Remendur reeds before and after glass/metal sealing

Experiment	Samples	Complete Reed—search coil on paddle		Shank		Paddle	
		Hc (oer.)	ϕ_R (maxwells)	Hc (oer.)	ϕ_R (maxwells)	Hc (oer.)	ϕ_R (maxwells)
A	610°C Aged reed	27.4 (3)	21.5 (3)	23.6 (3)	5.9 (3)	28.4 (3)	10.0 (3)
	610°C Sealed reed	29.7 (12)	19.3 (12)	29.0 (16)	6.7 (16)	28.7 (16)	10.0 (16)
B	Accelerated speed seal	28.3 (11)	21.6 (11)	25.9 (11)	6.1 (11)	28.9 (11)	10.3 (11)
C	Sealed reed—normal lamp power	29.5 (6)	21.1 (6)	28.2 (6)	6.4 (6)	29.2 (6)	10.3 (6)
	Sealed reed—reduced lamp power	28.0 (6)	22.0 (6)	24.9 (6)	6.1 (6)	29.1 (6)	10.2 (6)

(*i*) H applied = 300 oersteds.
(*ii*) Numbers in parentheses indicate sample size.

γ (FCC) precipitate and return to the two-phase $\alpha_1 + \alpha_2$ structure or all-α_2 structure are apparent in Fig. 5.

The results from the reeds sealed by using the accelerated speed sealing (Experiment B) and with reduced lamp power (Experiment C) show an improvement. As can be noted in Table II, the coercivity increase for the complete reed was smaller, and no drop in remanence occurred. The data on the dissociated shanks show that a coercivity increase still occurred on these shanks, but it was markedly smaller than that for the standard sealing operation. Metallographic results also showed that a structural change had taken place but was confined within a smaller percentage of the shank length. This probably accounts for the less drastically altered magnetic properties.

IV. SUMMARY AND CONCLUSIONS

The alteration of the magnetic properties of Remendur by the glass-to-metal sealing operation from those properties developed in the nominal 600°C-aging anneal is real. This is a consequence of excessive temperatures during the sealing operation causing a dissolving of the γ (FCC) precipitate and a reversion of the structure back to that developed by the typical 900 to 950°C strand anneal used in processing the wire. With the present time/temperature sealing cycle used in production, the following more specific conclusions can be listed:

(*i*) From one-half to two-thirds of the shank length, beginning at the paddle/shank transition region, is altered by the sealing operation.

(*ii*) A significant increase in coercive force occurs in the shank.

(*iii*) Laboratory results indicate that a significant drop in remanence also occurs during elevated temperature exposures, which are sufficiently high to cause a reversion to the two-phase $\alpha_1 + \alpha_2$ or all-α_2 structures.

(*iv*) Variations in the microstructure of 0.535-mm wire from the strand anneal have little effect on the subsequent structure and magnetic properties changes during sealing.

A reduced-temperature sealing cycle holds some promise for minimizing this change, but all 2.5- to 3.0-percent vanadium Remendur alloys are likely to exhibit this same behavior independent of vanadium content or prior aging heat treatment. If this properties change is considered unacceptable, a modification in the sealing cycle or a redesign of the contact to account for this change are the most promising approaches, since the materials response cannot be altered.

V. ACKNOWLEDGMENTS

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